AD-A057 313 LEHIGH UNIV BETHLEHEM PA DEPT OF MECHANICAL ENGINEE--ETC F/G 11/6
MECHANISM OF CORROSION FATIGUE OF STEEL AND TITANIUM ALLOYS.(U)
JUL 78 G W SIMMONS, R P WEI N00014-75-C-0543

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LEHIGH UNIVERSITY
Bethlehem, Pennsylvania 18015

Department of
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(11) 3 Jul [redacted] 78

(10) G. W. Simmons
R. P. Wei

(12) 16p.

Dr. P. A. Clarkin, Code 471
Metallurgy Program
Office of Naval Research
Department of the Navy
Arlington, VA 22217

Subject: (9) Semi-Annual Report for period ending 30 Jun 78

Reference: ONR Contract (15) N00014-75-C-0543, NR 036-097
(6) "Mechanism of Corrosion Fatigue of Steel and Titanium Alloys."

Dear Phil:

We are submitting herewith an informal report of progress for the six months ending June 30, 1978. The principal goal of research under the referenced contract is to characterize and understand environment-enhanced subcritical-crack growth (i.e., stress corrosion cracking and corrosion fatigue) in structural materials as a basis for developing rational methods for estimating serviceable lives of structural components. Both surface chemistry and fracture mechanics techniques are used in this research program. Efforts during this period were directed primarily in two areas: (a) crack growth and surface reaction studies of the Ti-5Al-2.5Sn alloy, and (b) study of the influence of water vapor pressure on fatigue crack growth in a 2219-T851 aluminum alloy. (The second area of study received partial support from this contract, and from unsolicited grants from the ALCOA Foundation to G. W. Simmons and R. P. Wei.) Results from these efforts were reviewed with you during our visit with you on June 26, 1978. Brief summaries of the current status in each area are given here.

A 2.54 cm (1 in.) thick plate of Ti-5Al-2.5Sn alloy was obtained through the courtesy of the Air Force Materials Laboratory for use in this program. The material was produced under an AFML sponsored program to study titanium alloy scrap reclamation processes at RMI Company. Iron and oxygen levels are somewhat higher than those specified for the ELI grade, and the plate contained some tungsten carbide chips that were intentionally added (though thought to be completely removed) for the reclamation study. The plate has been re-heat treated by annealing in vacuum at 900°C (1650°F) for 1 hour and cooling in air to produce an equiaxed α phase microstructure. Texture determinations have been made (see Figure 1) and showed that there is some preferred orientation of the basal plane perpendicular to the transverse direction of the plate. For the chosen specimen orientation (TL), the preferred basal planes would be parallel to the crack plane, with the crack growth direction along $<10\bar{1}0>$.

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PLUS OR MINUS

The kinetics of subcritical-crack growth under sustained load in 3.5 pct NaCl solution have been determined over a range of temperatures from 3 to 90°C ~~(see Figure 2)~~. 0.64 cm (1/4 in.) thick wedge-opening-load (WOL) specimens were used. A well defined K-independent stage (Stage II) of growth was observed at each temperature, and the rate was controlled by a thermally activated process having an apparent activation energy of 20 ± 5 kJ/mol (on a 95 pct confidence level basis)~~, Figure 3~~. These Stage II rates were substantially higher than those reported by Williams and Nelson, at corresponding temperatures, for this alloy tested in hydrogen at 0.9 atm. The apparent activation energy for crack growth in these two environments, however, are statistically equivalent. No identification of the rate controlling process (or processes) has been made.

Attempts have also been made to produce sustained-load crack growth in water vapor and in distilled water. It was found that crack growth occurred only at very high K levels in these environments. Consequently, experiments have been initiated to examine the role of chloride ion concentration on crack growth in this Ti-5Al-2.5Sn alloy.

The kinetics of the reactions of oxygen and water vapor with Ti-5Al-2.5Sn alloy at room temperature have been studied using Auger electron spectroscopy (AES). Because of the relatively high rate of contamination of Ti-5Al-2.5Sn alloy surfaces by CO during ion etching, it was not convenient to use polished specimens. Clean surfaces produced by impact fracture in vacuum (at less than 10^{-9} torr) were used instead for these studies. Changes in the normalized oxygen Auger (510 ev) signal as a function of exposure to oxygen and to water vapor are shown in Figure 4. The initial rates of reaction of the surface with each of these gases are similar. The "saturation" coverage, however, is higher for oxygen than for water vapor. By assuming that the water vapor reaction rate is first order with respect to the available surface sites and that the initial sticking coefficient is unity, a "saturation" coverage of approximately 0.65×10^{15} cm^{-2} was determined from the experimental data. This coverage is equal to one-half the density of surface atoms in a polycrystalline titanium specimen. The "saturation" coverage for the reaction of oxygen would be equal to the density of surface atoms, if one assumes that there is no appreciable attenuation of the oxygen Auger electrons from this layer. Apparently, therefore, the "saturation" coverage for the reaction of water vapor with Ti-5Al-2.5Sn alloy corresponds to one-half of a monolayer, and that for the reaction with oxygen, a full monolayer or possibly a single layer of oxide.

Studies are planned to determine whether there is any further reaction beyond these "saturation" coverages at higher exposures, and to determine with ESCA the changes in oxidation state of titanium as a function of exposure. Studies of the temperature dependence of these reactions will be made for correlation with the crack growth experiments. Studies of the influence of chloride ions on the kinetics of water vapor reaction will also be made.

Fracture mechanics and surface chemistry studies were carried out to develop further understanding of the influence of water vapor on fatigue crack growth in aluminum alloys. The room temperature fatigue crack growth response was determined for 2219-T851 aluminum alloy (1.65 cm or 0.65 in. thick plate) exposed to water vapor at pressures from 7.5×10^{-3} to 0.2 torr over a range of stress intensity factors. Data were also obtained in vacuum (at about 2×10^{-9} torr), air (at 40 to 60 pct

relative humidity), distilled water, and 3.5 pct NaCl solution. The test results (see Figure 5 to 7) showed that, at a frequency of 5 Hz, the rate of crack growth is essentially unaffected by water vapor until a threshold pressure is reached. The rates then increased and reached a maximum within one order of magnitude increase in vapor pressure from this threshold. The maximum rate is equal to that obtained in air, distilled water, and 3.5 pct NaCl solution. The transition range, in terms of pressure/frequency, is identical to that reported by Bradshaw and Wheeler on another aluminum alloy.

Parallel studies of the reactions of oxygen and water vapor with clean surfaces of 2219-T851 aluminum alloy were made by Auger electron spectroscopy. Similar to the case of the titanium alloy, the clean surfaces were produced by in situ impact fracture at pressures below 10^{-9} torr. Changes in the normalized oxygen Auger (510 eV) signal as a function of exposure to oxygen and to water vapor are shown in Figure 8. The initial rates of reaction with water vapor appear to be faster than those for oxygen; (although the possibility of induced reaction with water vapor by the incident electron beam remains to be established). The reactions with water vapor reached "saturation" following about 2×10^{-5} torr-s exposure. After a relatively fast initial reaction with oxygen, on the other hand, the reaction continues at a slower rate and does not reach "saturation" following exposure up to 5×10^{-3} torr-s. Further analyses of these data and additional experiments are in progress.

Comparison of the surface reaction data (Figure 8) with the data on fatigue crack growth response (Figure 7) shows a similar trend with increasing water vapor exposure. If pressure/frequency (p/f) is used as a measure of exposure in the fatigue experiments, then the transition range for fatigue response appears to be up to 10^3 times higher than that for the surface reactions. Correlation is therefore not straightforward, and may involve pressure attenuation at the crack tip. This possibility is suggested by differences in fracture surface appearance between the near-surface and mid-thickness regions of specimens tested within the transition region (i.e., between 0.01 and 0.1 torr). Further investigations to resolve this problem are in progress. Nevertheless, the difference in reaction rates, with water vapor, between 2219-T851 aluminum alloy and AISI 4340 steel (of about 10^8) would account for the previously reported differences in fatigue crack growth response in aqueous environments between aluminum and ferrous alloys. The fact that no further increase in the rate of fatigue crack growth was observed in the aqueous environments over that in water vapor at "saturation" is consistent with the observation that the water-aluminum reaction is limited. It suggests that enhancement of crack growth results from hydrogen produced during this reaction, rather than by anodic dissolution.

Presentations and Publications

G. W. Simmons - Seminar on "Application of AES and ESCA for Characterizing Surfaces of Materials", presented at CARTECH, Reading, PA, GTE-Sylvania, Seneca Falls, NY, and Air Products, Trexlertown, PA.

G. W. Simmons - "Application of Scanning Transmission Electron Microscopy for Characterizing Methanol Synthesis Catalysts", Meeting of the Pittsburgh Catalysis Society, Pittsburgh, PA, April 28, 1978.

G. W. Simmons - "Fracture Mechanics and Surface Chemistry Studies of Steel in Coal Gasification Systems", DOE/EPRI/NASA Workshop on Hydrogen Attack, Lehigh University, May 15-16, 1978.

R. P. Wei - "Nonsteady-State Phenomena Associated With Fatigue Crack Growth in Aggressive Environments", ASTM Workshop on Current Practices for Fatigue Crack Growth Rate Testing in Aggressive Environments, Washington, D. C., March 17, 1978.

R. P. Wei - "On Understanding Environment Enhanced Fatigue Crack Growth - A Perspective View (1968-1977)", ASTM Symposium on Fatigue Mechanisms, Kansas City, MO, May 22-23, 1978.

"Determination of Quantitative Sputter Rates of Iron Oxide by Auger Electron Spectroscopy (AES) and Ellipsometry", R. G. Hart and G. W. Simmons, J. Vac. Sci. Technology, 15, 714 (1978).

"In Situ Studies of the Passivation of Cobalt by Emission Mössbauer Spectroscopy", G. W. Simmons, to be published in the Proceedings of the Fourth International Symposium on Passivity, 1978.

"Fracture Mechanics and Surface Chemistry Studies of Subcritical Crack Growth in AISI 4340 Steel", G. W. Simmons, P. S. Pao and R. P. Wei, Met. Trans. A, to be published Aug., 1978.

"Environment Enhanced Fatigue Crack Growth in High-Strength Steels", R. P. Wei and G. W. Simmons: Stress Corrosion Cracking and Hydrogen Embrittlement of Iron Based Alloys, J. Hochmann, J. Slater, and R. W. Staehle, eds., NACE, 1978.

"Time-Dependent Flow of Solute Atoms Near a Crack Tip", Y. T. Chou, R. S. Wu, and R. P. Wei, Scripta Met., Vol. 12, (1978), pp. 249-254.

"Normal Surface Displacement Around a Circular Hole by Reflection Holographic Interferometry", P. M. de Larminat and R. P. Wei, Experimental Mechanics, Vol. 18, (1978), pp. 74-80.

"Fracture Mechanics Approach to Fatigue Analysis in Design", R. P. Wei, J. Eng'g. Mat'. & Tech., Vol. 100, (1978), pp. 113-120.

"Fractographic Analysis of Gaseous Hydrogen Induced Cracking in 18Ni Maraging Steel", R. P. Gangloff and R. P. Wei: Fractography in Failure Analysis, ASTM, STP 645, 1978.

"Effect of Frequency on Fatigue Crack Growth Response of AISI 4340 Steel in Water Vapor", P. S. Pao, W. Wei and R. P. Wei, Proceedings of Symposium on Environment Sensitive Fracture of Engineering Materials (TMS-AIME, 1977), to be published.

"The Combined Influence of Chemical, Metallurgical and Mechanical Factors on Environment Assisted Cracking", D. P. Williams, III, P. S. Pao and R. P. Wei, Proceedings of Symposium on Environment Sensitive Fracture of Engineering Materials (TMS-AIME, 1977), to be published.

"Effect of Measurement Precision and Data-Processing Procedures on Variability in Fatigue-Crack Growth-Rate Data", R. P. Wei, W. Wei and G. A. Miller, Submitted for publication to J. of Test. & Eval., ASTM (1978).

"On Understanding Environment Enhanced Fatigue Crack Growth - A Perspective View (1968-1977)", R. P. Wei, ASTM Symposium on Fatigue Mechanisms, to be published by ASTM, 1978.

We are planning to present our results on the Ti-5Al-2.5Sn alloy and the 2219-T851 aluminum alloy at the TMS-AIME Fall Meeting in St. Louis. Copies of abstracts that we have submitted to TMS are enclosed for your information.

Best regards,

Sincerely yours,

Bob

R. P. Wei
Professor of Mechanics

RPW:ss
Encls.
cc: Mr. Sidney Brown - ONR Phila.
Dr. G. W. Simmons

FIGURE CAPTIONS

Figure 1: $\{10\bar{1}0\}$ pole figure for Ti-5Al-2.5Sn alloy. (Numbers represent multiples of random intensity. RD = rolling direction, TD = transverse direction, and ND = normal to plate surface.)

Figure 2: Kinetics of sustained-load crack growth for Ti-5Al-2.5Sn alloy tested in 3.5 pct NaCl solution. Specimens orientation: TL.

Figure 3: Influence of temperature on Stage II crack growth in Ti-5Al-2.5Sn alloy tested in 3.5 pct NaCl solution.

Figure 4: Kinetics of reactions of Ti-5Al-2.5Sn alloy with oxygen and with water vapor at room temperature.

Figure 5: Influence of water vapor pressure on the kinetics of fatigue crack growth in 2219-T851 aluminum alloy at room temperature.

Figure 6: Kinetics of fatigue crack growth for 2219-T851 aluminum alloy in air, distilled water and 3.5 pct NaCl solution at room temperature.

Figure 7: Influence of water vapor pressure (or pressure/frequency) on fatigue crack growth rate in 2219-T851 aluminum alloy at room temperature and $K_{max} = 10$ ksi$\sqrt{in}$.

Figure 8: Kinetics of reactions of 2219-T851 aluminum alloy with oxygen and with water vapor at room temperature.

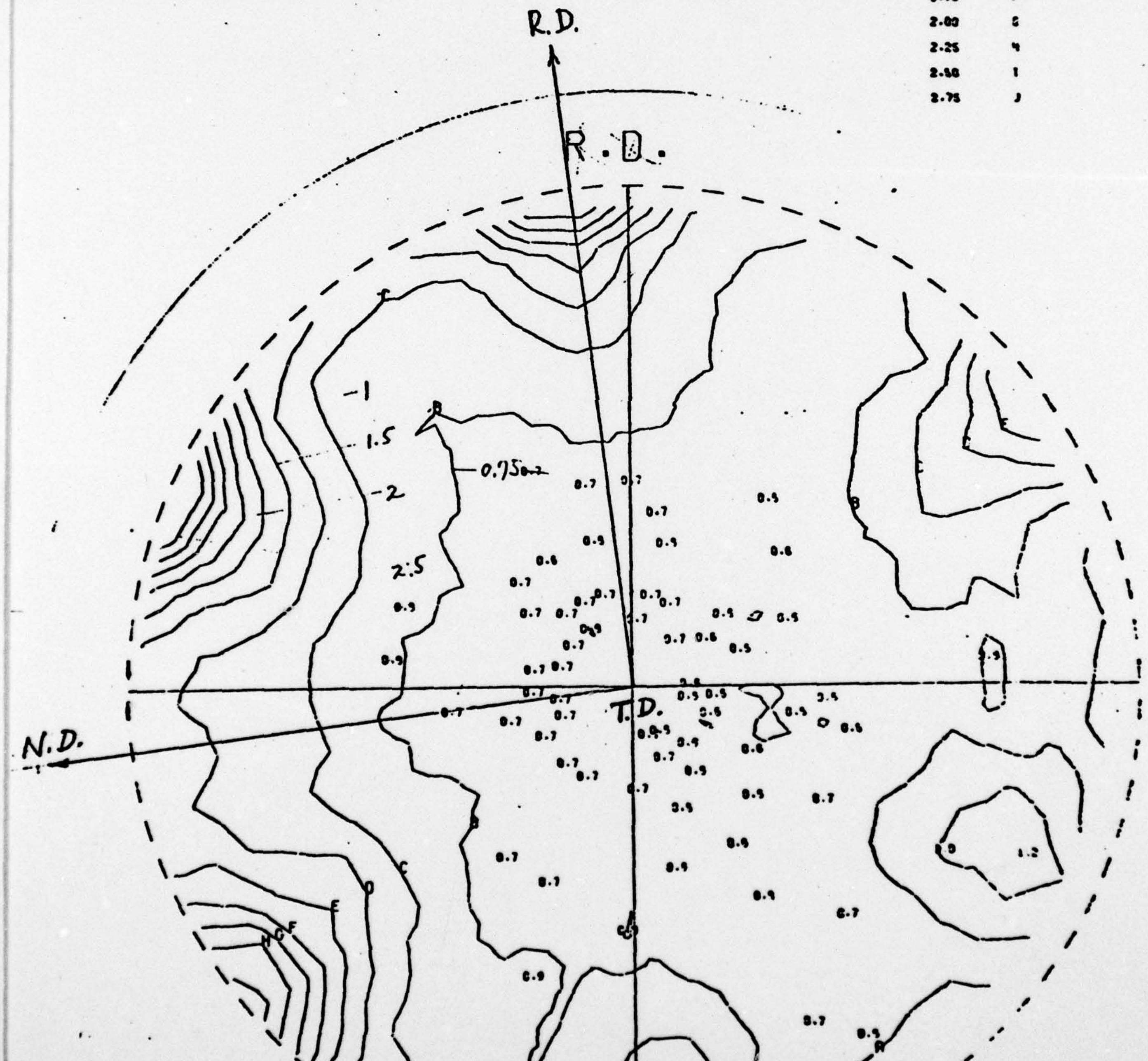
01/16/78 R.E.S. **Figure 1**

WHH POLE FIGURE DATA FOR SPEC TI-T (10$\bar{1}$0)REFL 1/16/78

MO RAD 50KV 20MA 1DEG SLIT MR REC SLIT

CONTOUR VALUES

0.50	A
0.75	B
1.00	C
1.25	D
1.50	E
1.75	F
2.00	G
2.25	H
2.50	I
2.75	J



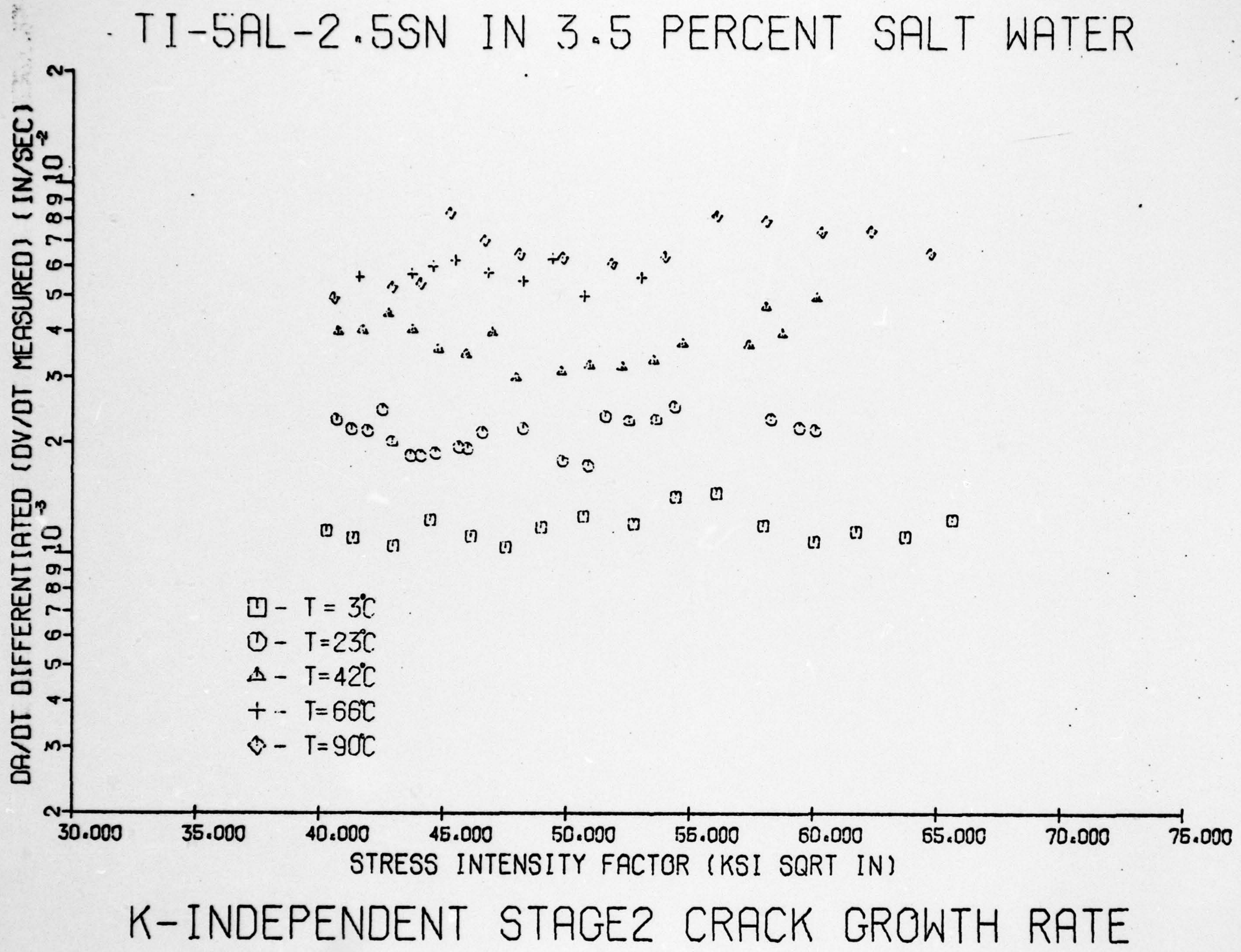

TI-5AL-2.5SN IN 3.5 PERCENT SALT WATER
DA/DT DIFFERENTIATED (DV/DT MEASURED) (IN/SEC)
2
3
4
5
6
7
8
9
10^-3
2
3
4
5
6
7
8
9
10^-2
2
30.000
35.000
40.000
45.000
50.000
55.000
60.000
65.000
70.000
75.000
STRESS INTENSITY FACTOR (KSI SQRT IN)
- T = 3°C
- T=23°C
- T=42°C
- T=66°C
- T=90°C
K-INDEPENDENT STAGE2 CRACK GROWTH RATE

Figure 2

Figure 3

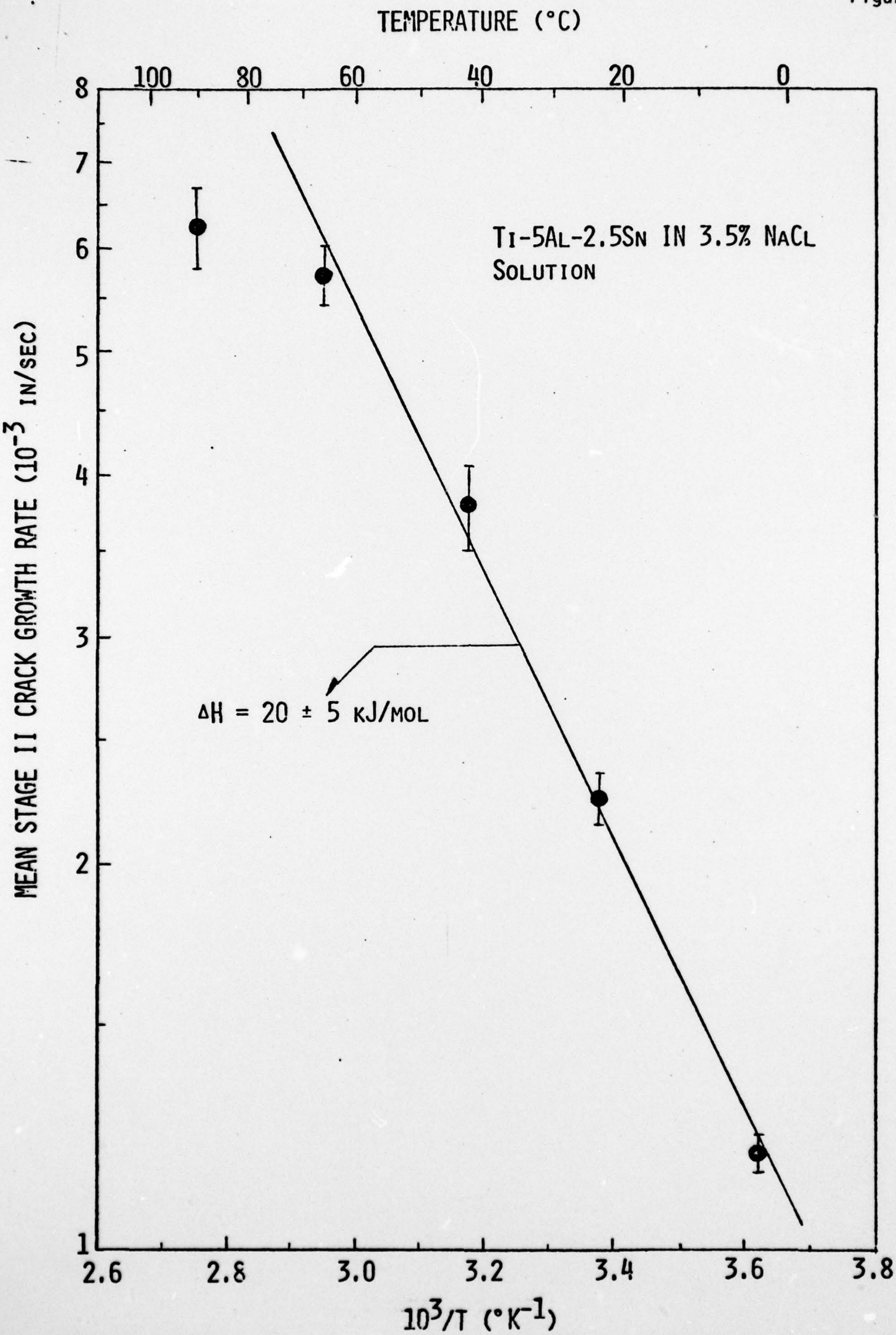
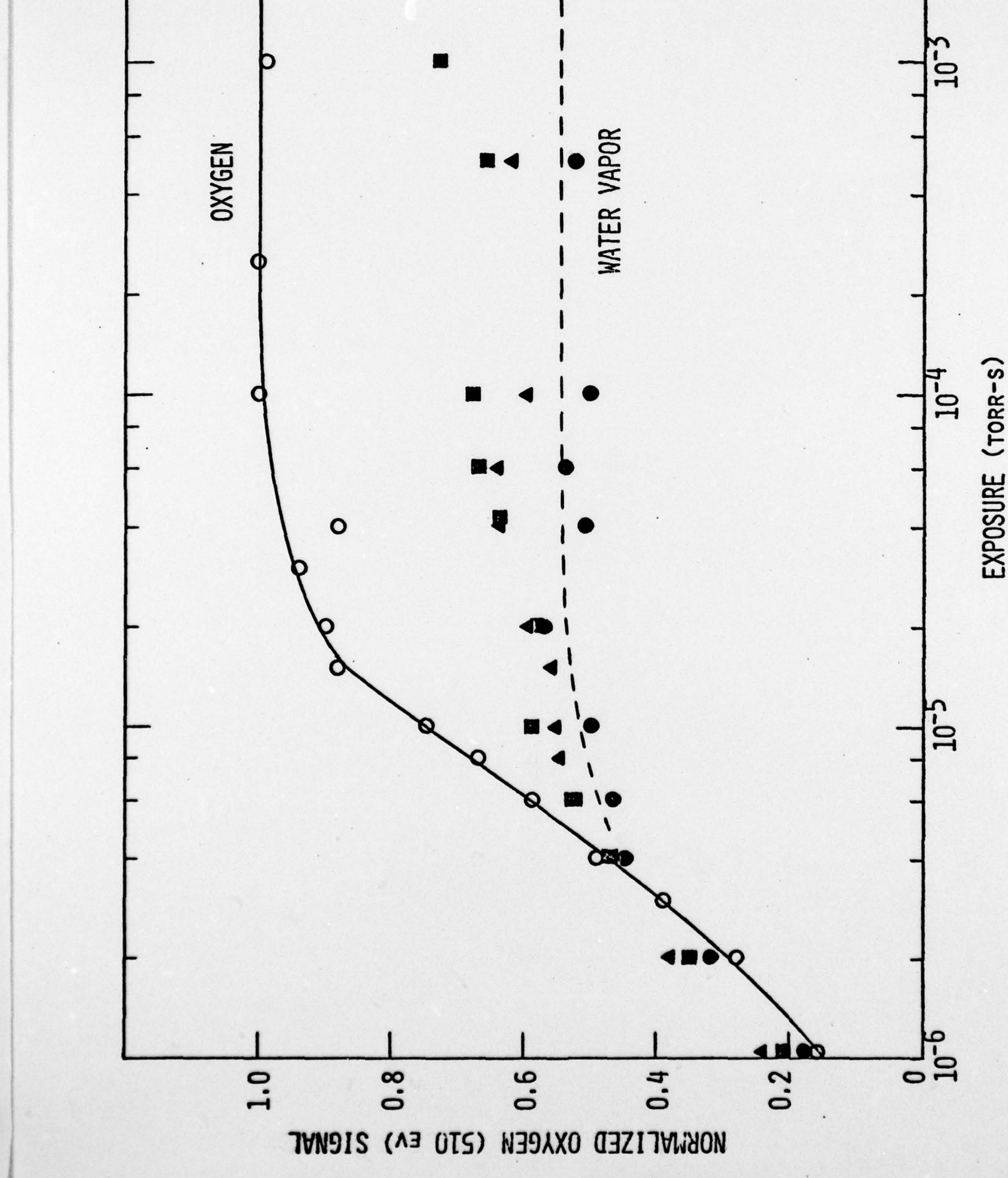
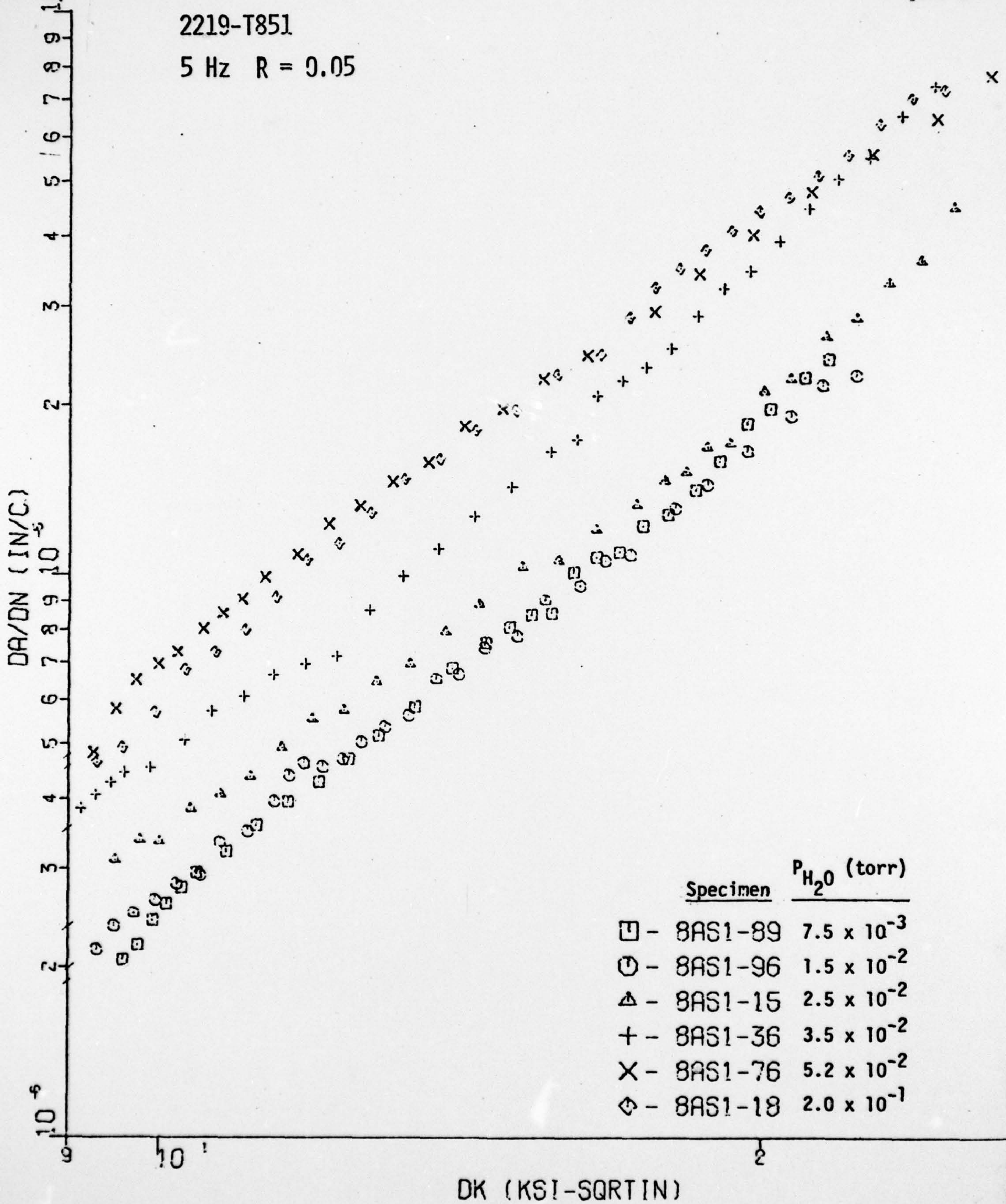

TEMPERATURE (°C)
100
80
60
40
20
0
8
7
6
5
4
3
2
1
MEAN STAGE II CRACK GROWTH RATE (10^{-3} IN/SEC)
Ti-5Al-2.5Sn IN 3.5% NaCl
Solution
ΔH = 20 ± 5 kJ/mol
2.6
2.8
3.0
3.2
3.4
3.6
3.8
$10^3/T$ (°K^{-1})

Figure 4



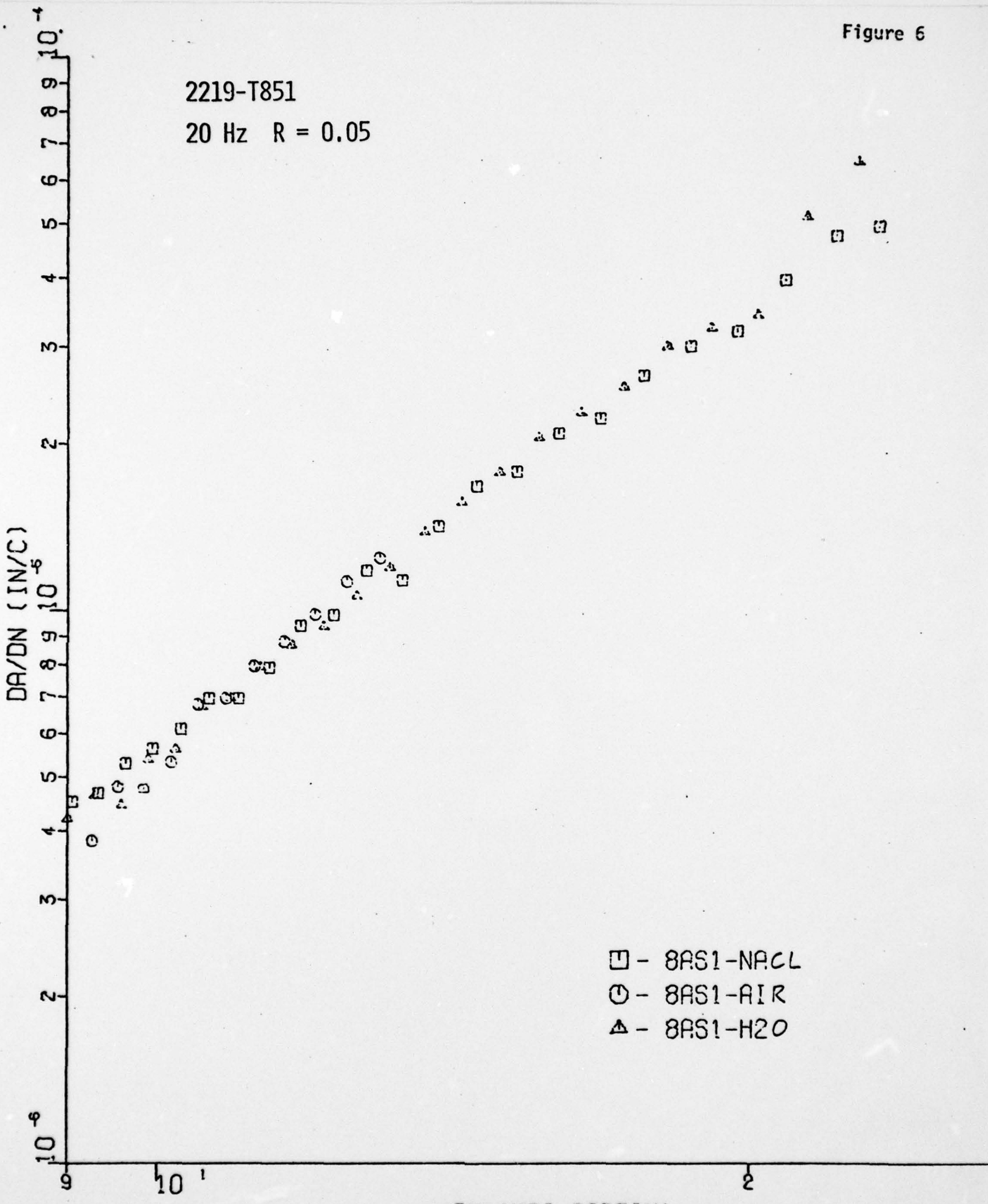
OXYGEN
WATER VAPOR
EXPOSURE (TORR-s)
NORMALIZED OXYGEN (510 eV) SIGNAL
0
0.2
0.4
0.6
0.8
1.0
10^{-6}
10^{-5}
10^{-4}
10^{-3}

Figure 5



FATIGUE CRACK GROWTH

Figure 6



FATIGUE CRACK GROWTH

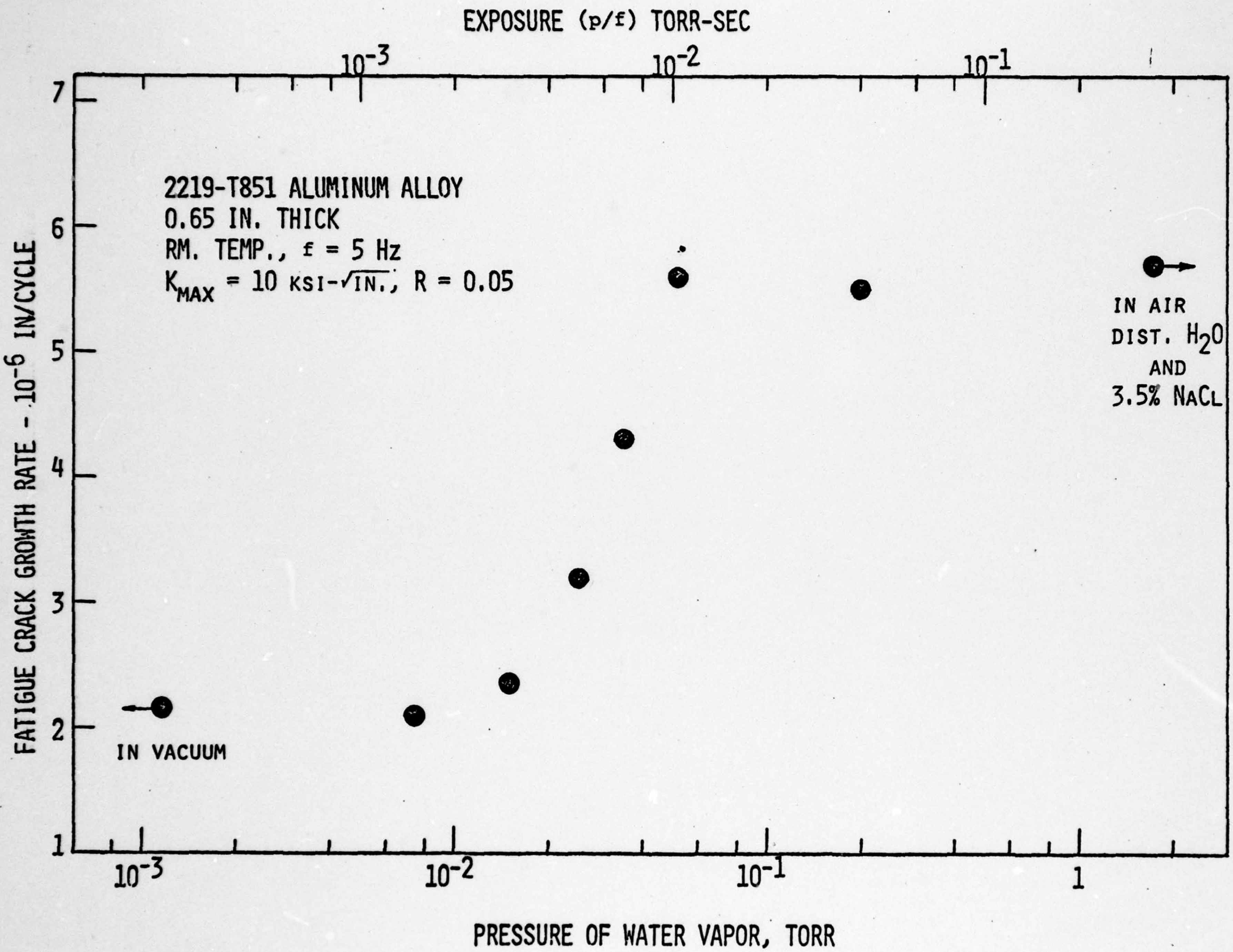
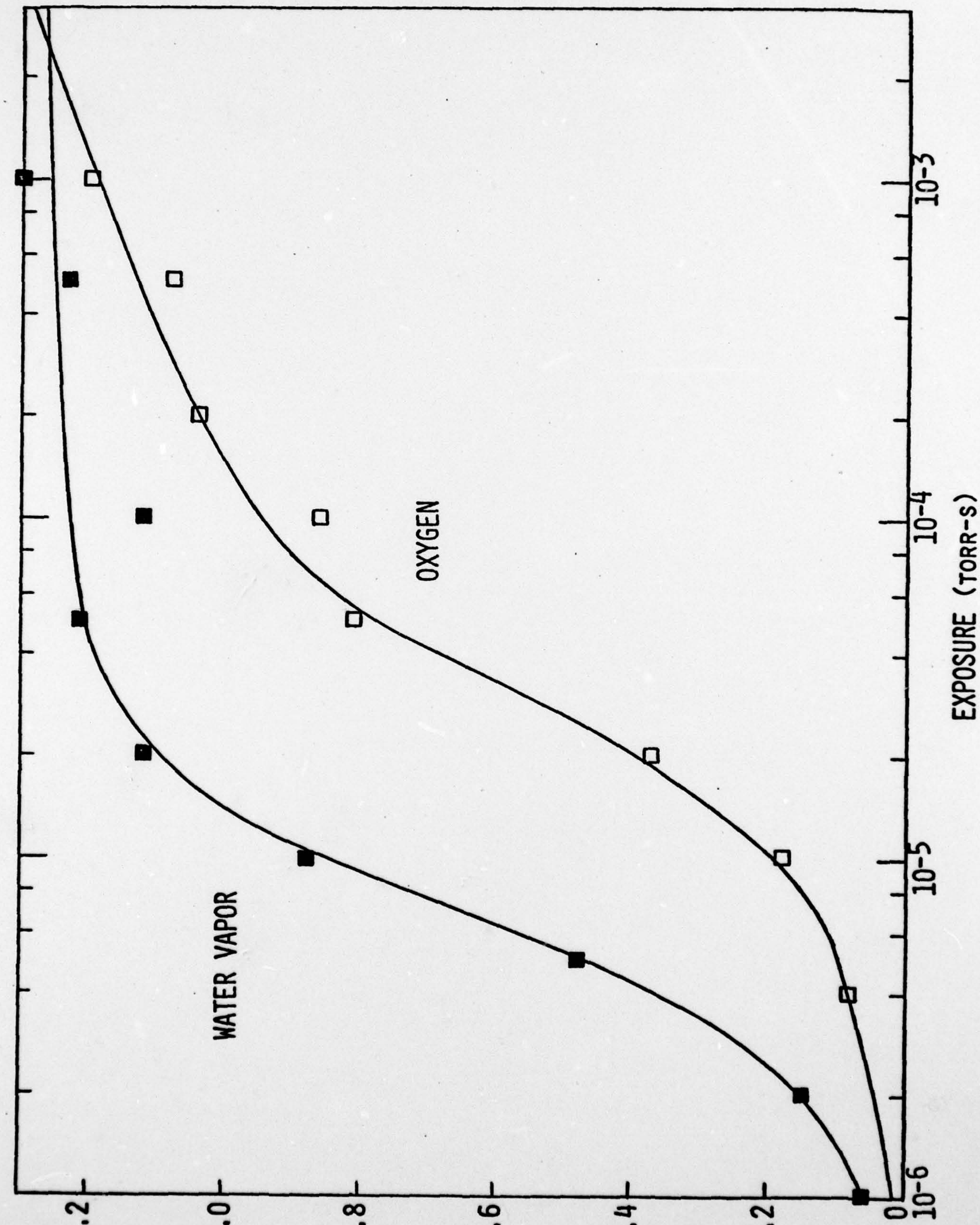

EXPOSURE (p/f) TORR-SEC
10^{-3}
10^{-2}
10^{-1}
2219-T851 ALUMINUM ALLOY
0.65 IN. THICK
RM. TEMP., f = 5 Hz
K_{MAX} = 10 KSI-$\sqrt{IN.}$, R = 0.05
IN AIR
DIST. H_2O
AND
3.5% NACL
IN VACUUM
FATIGUE CRACK GROWTH RATE - 10^{-6} IN/CYCLE
1
2
3
4
5
6
7
10^{-3}
10^{-2}
10^{-1}
1
PRESSURE OF WATER VAPOR, TORR

Figure 7

Figure 8



NORMALIZED OXYGEN (510 eV) SIGNAL
0
0.2
0.4
0.6
0.8
1.0
1.2
EXPOSURE (TORR-S)
10-6
10-5
10-4
10-3
WATER VAPOR
OXYGEN

x x

6. Fracture Titanium Alloy Stress Corrosion Cracking
Fracture Mechanics

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STRESS CORROSION CRACKING OF Ti-5Al-2.5Sn ALLOY IN AQUEOUS ENVIRONMENTS:* G. Shim, P. S. Pao and R. P. Wei, Lehigh University, Bethlehem, PA 18015

The kinetics of sustained-load subcritical crack growth for a Ti-5Al-2.5Sn alloy in 3.5% NaCl solution were determined over a range of temperatures from 3° to 90°C. Crack-tip stress intensity factor (K) was used as a measure of the crack driving force. A well defined K-independent stage (Stage II) was identified in each test, and the rate was controlled by a thermally activated process having an apparent activation energy of 20 ± 5 kJ/mol. The Stage II rates were substantially higher than rates reported by Williams and Nelson for this alloy tested in hydrogen at 0.9 atm. The activation energies for Stage II crack growth in these two environments, however, were statistically equivalent. The effect of chloride-ion concentration on the room temperature crack growth kinetics was also studied. Stage II rates were found to be only slightly affected by change in chloride-ion concentration. These results will be discussed in relation to the possible cracking mechanisms.

*Research sponsored in part by the Office of Naval Research under Contract N00014-75-C-0543, NR036-097.

x x

6. Fracture / 10. Surfaces Aluminum Alloy Corrosion Fatigue
Fracture Mechanics Oxidation Auger Spectroscopy

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FRACTURE MECHANICS AND SURFACE CHEMISTRY STUDIES OF FATIGUE CRACK GROWTH IN AN ALUMINUM ALLOY:* P. S. Pao, R. G. Hart, G. W. Simmons and R. P.Wei, Lehigh University, Bethlehem, PA 18015

Fracture mechanics and surface chemistry studies were carried out to develop further understanding of the influence of water vapor on fatigue crack growth in aluminum alloys. The room temperature fatigue crack growth response was determined for 2219-T851 aluminum alloy exposed to water vapor at pressures from 1 Pa to 30 Pa over a range of stress intensity factors (K). Data were also obtained in vacuum (at 0.25 μPa) and in distilled water. The test results showed that, at a frequency of 5 Hz, the rate of crack growth is essentially unaffected by water vapor until a threshold pressure is reached. Fatigue crack growth rates then increased and reached a maximum within one order of magnitude increase in vapor pressure from this threshold. The maximum rate is equal to that obtained in distilled water. Parallel studies of the reactions of water vapor with fresh alloy surfaces (produced *in situ* by impact fracture) were made by Auger electron spectroscopy. The extent of surface reaction, monitored by changes in the oxygen Auger signal, showed a similar trend with increasing water vapor exposure. Correlation between the fatigue crack growth response and surface reaction kinetics will be discussed.

*Research sponsored in part by the Office of Naval Research under Contract N00014-75-C-0543, NR036-097, and by the Alcoa Foundation.